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Extraction of passion fruit seed oil using supercritical CO_2: a study of mass transfer and rheological property by Bayesian inference

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RESUMEN

Extracción de aceite de semillas de frutos de la pasión con CO_2 supercrítico: estudio de transferencia de masa y propiedades reológicas mediante deducción Bayesiana

Se ha estudiado la extracción de aceite de semillas de frutos de la pasión con CO_2 supercrítico. Los datos experimentales se obtuvieron para una extracción llevada a cabo a 15, 20 y 25 MPa; a temperaturas de 40 y 50 °C y a flujos de CO_2 de 1,5 y 3,0 mL min^{-1}. Un incremento de la presión, la temperatura y del flujo de CO_2 aumentó el rendimiento. El máximo rendimiento de la extracción obtenida fue de 18,5%. Los coeficientes de transferencia de masa para el aceite de fruta de la pasión encontrados fueron 8,496 × 10-5 s^{-1} a 25 MPa, 50 °C y 3 mL s^{-1} de flujo de CO_2. Se observó un comportamiento de fluido dilatante en todos los ensayos reológicos.

PALABRAS CLAVE: Aceite de semillas – CO_2 supercrítico – Frutos de la pasión – Inferencia bayesiana.

SUMMARY

Extraction of passion fruit seed oil using supercritical CO_2: a study of mass transfer and rheological property by Bayesian inference

The extraction of oil from passion fruit seeds using supercritical CO_2 was studied. Experimental data were obtained for extraction conducted at 15, 20 and 25 MPa; at temperatures of 40 and 50 °C with CO_2 flow rates of 1.5 and 3.0 mL min^{-1}. An increase in the pressure, temperature and CO_2 flow rate increased the yield. The maximum extraction yield obtained was 18.5%. The mass transfer coefficients for passion fruit oil were found to be 8.496 × 10-5 s-1 at 25 MPa, 50 °C and 3 mL s^{-1} CO_2 flow rate. Dilatant fluid behavior was observed in all tests of the rheological study.

KEY-WORDS: Bayesian inference – Passion fruit – Seed oil – Supercritical CO_2.

1. INTRODUCTION

Brazil is the world's largest producer of passion fruit. The yellow passion fruit (*Passiflora edulis f. flavicarpa*) represents about 97% of the planted area of passion fruit in Brazil. An estimated 60% of the Brazilian produce is sold in producer markets/supermarkets and consumed *in natura.* The remainder is processed by industry into juice as a main product. Passion fruit seeds are usually discarded as a byproduct after extraction of the juice from the fruit. However, these seeds are a good source of oil which contains unsaturated fatty acids, such as oleic acid (C18:1) and linoleic acid (C18:2) and finds application in the food and especially the perfume and aroma industries (Lopez, 1980; Morton, 1987; Nyanzi *et al.*, 2005; Shucheng *et al.*, 2008).

The commercialization of vegetable oils has proven to be highly beneficial for the food, cosmetic and pharmaceutical industries (Temelli, 2009). In this context, an adequate oil extraction process is required; keeping in in mind the environmental effects of the process without compromising the final product quality. Vegetable oil is conventionally extracted using the mechanical press process followed by liquid organic solvents in the extraction and solvent recuperation by distillation. The mechanical processes bring about thermal degradation of the active components and the use of solvents causes solvent contamination. To combat these detrimental effects, several alternative methods for oil extraction have been studied around the world. One of the excellent alternative methods, which minimizes the use of liquid organic solvents and facilitates solvent recovery, is the use of pressurized fluids such as supercritical CO_2 (SC-CO_2). The extraction with pressurized fluids permits the efficient removal of triglycerides from vegetable oils, enables easy separation of solvent, oil and residue of the process, resulting in products with no residual solvents (Temelli, 2009; Acosta *et al.,* 1996; Gracia *et al.,* 2009; Herrero *et al.,* 2010, Reverchon 1997). The SC-CO_2 extraction process has become the focus of attention in recent years due to its chemical and physical properties: non-flammable, non-toxic, non-corrosive, etc. Furthermore, the extracted product is of good quality and scarcely requires further refining. Thus, SC-CO_2 technology has been applied to the extraction of oil from a large number of materials over the last twenty years and shows a bright future.

This work reports the effect of extraction pressure, flow rate and temperature on the SC-CO_2 extraction of passion fruit oil from seeds and a study of mass transfer and rheological property by Bayesian inference.

2. THEORY

2.1. Mass transfer behavior

Kinetics and theoretical models can describe the mass transfer behavior during SC-CO_2 extraction. According to Özkal *et al.,* (2005) the theoretical models include the analytical or numerical solutions of the governing mass transfer equations. The model proposed by Andrich *et al.,* (2001) has been extensively used to describe mass transfer behavior (Özkal *et al*., 2005; Adeib *et al*., 2010).

The kinetics model by Andrich *et al.* (2001) was used in this work to describe the mass transfer as:

$$\frac{dm_e}{dt} = k\left(m_{s,t} - m^*_{s,t}\right) \qquad \text{Eq.(1)}$$

where: m_e is the amount of oil extracted in grams at time t; *k* is the mass transfer coefficient in s^{-1}; $m_{s,t}$ is the amount of unextracted oil at time t; $m^*_{s,t}$ is the amount of unextracted oil at time *t* if equilibrium between two phases has been reached.

Supposing, $m^*_{s,t}$, is negligible, because pure solvent is continuously fed to the extractor, and $m_{s,t}$ is equal to the difference between oil initially present in the sample ($m_{s,0}$) and oil extracted at time t, than Eq.(1) becomes:

$$\frac{dm_e}{dt} = km_{s,t} = \left(m_{s,0} - m_e\right) \qquad \text{Eq.(2)}$$

Integrating Eq. 2, it gives

$$ln\left(\frac{m_{s,0}}{m_{s,0} - m_e}\right) = kt \qquad \text{Eq.(3)}$$

The slope of the straight line passing through the origin of the axes t and $\left(\frac{m_{s,0}}{m_{s,0} - m_e}\right)$ gives k and the amount of oil extracted at time t is,

$$m_e = m_{s,0}\left(1 - e^{-kt}\right) \qquad \text{Eq.(4)}$$

The extraction yield in g oil/g seed sample was calculated by dividing the amount of oil extracted (Eq. (4)) by the amount of sample.

2.2. Bayesian inference

In general, the study of modeling SC-CO_2 extraction and rheological behavior have been carried out by a frequentist approach, adjusting nonlinear models, which aim to synthesize pieces of information into parameter estimates to be interpreted (Papamichail *et al.,* 2000; Reverchon 1997; Souza *et al.,* 2008; Meziane *et al.,* 2006; Fiori, 2007; Han *et al.,* 2009; Ixtaina *et al.,* 2010; Corso *et al*., 2010; Saxena *et al.,* 2011). According to Oliveira *et al.,* (2012a)(2012b), the estimation is based on iterative processes such as that of Gauss-Newton, DUD and Marquardt algorithm, due to the nonlinearity of variables. Such procedures minimize the sum of residue squares. However, when individual adjustments are considered, i.e. adjustments for many experimental units of mathematically complex models or there are few possible longitudinal observations, interactive methods frequently provide negative estimates for parameters. This can cause the formation of atypical curves.

Furthermore, regarding comparisons of curves deriving from different treatments, the distribution of nonlinear model parameter estimators does not usually follow the Gaussian distribution. Therefore, the process to formulate statistical tests becomes complex if enough attention is not paid to presuppositions related to the asymptotic theory (Oliveira *et al.,* 2012). Bayesian inference, involving the adjustment of linear and nonlinear regression models, was successfully used in recent years, as it reduced the number of biased estimations even when little information was used (Oliveira *et al.* (2009); Oliveira *et al.* (2011); Oliveira *et al.* (2012a, 2012b).

3. MATERIALS AND METHODS

3.1. Sample preparation

Passion fruit seeds used in the experiments were kindly provided by Polpa Norte (Paraná, Brazil). Initially, the seeds were dried in a circulating air oven (Nova Ética, model 400/4ND, Brazil) at 353K for 72 h. The dried seeds were milled using a knives mill (Framo-Geratetechnik, model A 70, Germany) to produce particles of a mean diameter of 778 µm.

A seed lot sample was physico-chemically characterized based on moisture, fixed mineral residue, raw fiber, total lipids, and crude protein – according to the procedures of the BRASIL - Adolfo Lutz Institute (2008). Analyses were performed in triplicate and their mean values are presented in this study.

3.2. Supercritical fluid extraction procedures

All experiments were performed in a laboratory scale unit, as described by Souza *et al.* (2008), which basically consists of a solvent reservoir (CO_2, White Martins S.A. with 99.9% of purity), two thermostatic baths, a syringe pump (ISCO, Model 500D) and an extractor with an internal volume of approximately 150 mL (diameter of the bed is 2.52 cm, and height of the bed is 29 cm). In each experiment, the extractor was loaded with approximately 15 g of powdered sample. The bed was filled with passion fruit crushed seed powder in a random manner. After the column temperature and pressure had been stabilized, the system was kept in contact with the passion fruit seeds for at least 1 h to facilitate system stabilization. Next, the CO_2 was pumped into the bed of passion fruit seeds at a constant flow rate. The temperature (T), pressure (P) and flow rates (F) were the independent variables studied in the oil yield (Y). The SC-CO_2 extraction experiments were conducted at temperatures of 313 and 323 K, pressures of 15, 20 and 25 MPa and flow rate of 1.5 and 3 cm^3 min^{-1}. After a pre-established period (10 minutes), extraction was interrupted for the measurement of the extracted mass. The experiments were carried out for 200 minutes of extraction, isothermally at constant pressure and flow rate. Runs were duplicated for all conditions.

3.3. Classical extraction

In order to determine the amount of oil in the passion fruit seeds, exhaustive extractions were performed in a Soxhlet extractor (Tecnal, Brazil). Approximately 5 g of passion fruit seeds, prepared as described in Section 3.1, were extracted in Soxhlet for 24 h according to the procedure described by BRASIL - Instituto Adolfo Lutz (2008), at the boiling point of hexane.

3.4. Oil yield

In all experiments carried out in this work the yield (Y) from extraction was calculated using the following equation:

$$Y(\%) = 100 \cdot \frac{W_O}{W_s} \qquad \text{Eq. (05)}$$

where w_O denotes oil content extracted and w_s indicates the seed content used in extraction.

3.5. Physical and chemical properties of seed oil

Oil from the seeds was subjected to physical and chemical characterization, as proposed by Liu *et al.,* (2009). The color of oil at room temperature was noted by visual inspection, while the density was determined by the AOAC methodology 985.19. The refractive index was determined at room temperature. Measurements of various chemical parameters such as acid value (AOAC 969.17), peroxide value (AOAC 965.33), saponification number (AOAC 920.160), non-saponification matter (AOAC 933.08), iodine value (AOAC 993.20), insoluble impurities (AOCS Ca 3a-46) and moisture and volatile matter (AOAC 926.12) were carried out following the official methods of analysis by AOAC (1990). Each oil sample was analyzed in triplicate and their mean values are reported in this work.

3.6. Rheological behavior

The viscosity of the passion fruit oil extracted at the best condition of SC-CO_2 was determined for the temperatures in a range of 10 to 70 °C in steps of 10 °C and the shear rates from 1.02 to 70 s^{-1}. A Brookfield Programmable Rheometer-Model DV-III, with concentric cylinders (spindle SC4-21, diameter = 17.48 cm, length = 31.72 cm, container diameter = 22 mm) was used. The dependence of oil viscosity on shear rate was investigated using the power law empirical models as follows:

$$\eta = K\gamma^n \qquad \text{Eq. (06)}$$

where η is the apparent viscosity (Pa.s), γ is the shear rate (s^{-1}), K is the consistency index (Pa.s^n) and n is the flow behavior index.

3.7. Mass transfer modeling

For the first stage it was assumed that the amount of oil extracted at time t (m_e) has normal distribution, i.e:

$$m_e \sim \mathrm{N}\left(\mathrm{f}(\mu,\mathrm{t}),\sigma^2\right).$$

All the model parameters were considered *prior* as non-informative Gamma distribution, i.e:

$$\text{parameters} \sim \text{gamma}(10^3,10^3)$$

Posterior distributions of parameters were obtained by BRugs on R program (R Development Core Team). 11,000 samples were obtained by Monte Carlo Markov Chain (MCMC), from which 1,000 were discarded ("burn-in samples") to eliminate the effect of initial values. Convergence chains were verified by Convergence Diagnosis and Output Analysis - CODA program - by Geweke (1992) and Heibelberger and Welch (1983) criteria.

4. RESULTS AND DISCUSSION

4.1. Raw material and characterization

The conventional extraction method led to 26.4% (w/w) yield of passion fruit oil. Tables 1 and 2 present the physico-chemical characterization results of passion fruit seeds and the fatty acid profile, respectively. Liu *et al.,* (2009) reported 8.57% palmitic acid, 1.66% estearic acid, 16.25% oleic acid, and 72.695% linoleic acid in passion fruit oil extracted by supercritical carbon dioxide.

4.2. Extraction yield

Table 3 presents the experimental conditions for the extraction of passion fruit seed oil using

Table 1
Physical-chemical characterization of yellow passion fruit seeds

Physical-chemical analysis	(% w/w)
Moisture and volatile substances	0.02 ± 0.0001
Fixed mineral residue	10.9 ± 0.0001
Raw fiber	46.5 ± 0.09
Crude protein	13.2 ± 0.01
Total lipids	24.8 ± 0.07

Table 2
Fatty acid profile of yellow passion fruit seeds extracted by Soxhlet

Fatty acids	Concentration g 100 g^{-1}	Percentage
Palmitic acid (C16:0)	4.05	16.3
Stearic acid (C18:0)	1.30	5.20
Oleic acid (C18:1n9c)	5.13	20.7
Linoleic acid (C18:2n6)	14.3	50.8
Total fat	24.8	–
Saturated fats	5.34	21.5
Monounsaturated fats	5.13	20.7
Polyunsaturated fats	14.3	57.8
Trans fats	0.00	0.00

carbon dioxide as solvent. The extraction yield was calculated as the ratio of the mass of the oil extracted to the mass of raw material fed into the extractor. Figure 1 describes the mass transfer behavior during SC-CO_2 extraction. As can be inferred by Figure 1, the temperature and pressure (density) render positive effects on the yield.

The ANOVA of the results presented in Table 3 indicate that temperature, flow rate and pressure significantly affect ($p < 0.05$) the supercritical oil extraction. As the difference between the treatments was significant, it is interesting to evaluate the magnitude of these differences by multiple comparison tests. In this work, Tukey's test was used to evaluate differences between any two or among more than two treatment means. Table 4 gives the means of the groups (flow rate, temperature and pressure) evaluated at 5% probability. Means with equal letters are not significantly different ($p < 0,05$).

The effect of pressure on extraction was investigated at pressures of 15, 20 and 25 MPa. As expected, the extraction rate increases significantly with the increase in pressure. This result is similar to what other researchers working on oil extraction from seeds have reported (Sovová, 1994; Louli

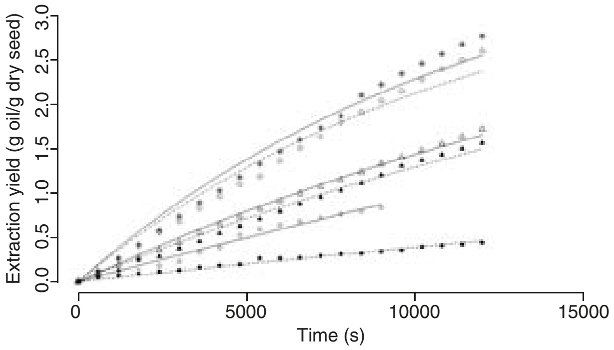

Figure 1
Effect of temperature and pressure on the extraction yield of passion fruit oil with time at a flow rate of 3.0 mL min^{-1} for different conditions.

et al., 2004; Salgin *et al.,* 2006; Jachmanián *et al.*, 2006; Fiori, 2007; Han *et al.*, 2009; Sánchez-Vicente *et al.,* 2009; Ixtaina *et al.*, 2010; Döker *et al.*, 2010; Corso *et al.*, 2010). This is attributed to the increase in the SC-CO_2 density, which results in the increase of its dissolving ability and the solubility of the components. With the pressure increasing from 15 to 25 MPa, the global mean extraction increased nearly 5 times and the extraction was more pronounced at 25 MPa.

The effect of temperature on the extraction was investigated at 40 and 50 °C. Figure 1 indicates that both the extraction rate and the total extraction yield at 40 °C are slightly higher than those at higher temperatures. The solubility of oil directly affects the extraction rate and this is controlled by the balance between the SC-CO_2 density and the oil vapor pressure. At high pressures, the influence of temperature on the solubility of oil is predominated by the oil vapor pressure effect, which increases the solubility of oil with an increase of temperature. While at low pressures, SC-CO_2 density has a pronounced effect on the solubility of oil and the solubility decreases with an increase in temperature (Kiriamiti *et al.,* 2001). In this case we assumed that our operational pressure is in the density effects predomination region. The effect of the CO_2 flow rate has also significantly affected the yield during passion fruit oil extraction. The extraction rate increased with an increase in the CO_2 flow rate. The dependence upon the flow rate indicates that the process is controlled by the solubility of the oil in solvent. According to Freitas *et al.*, (2008) the higher the temperature, the more favorable the solvent transport properties (viscosity and diffusion coefficient) become, making it easier for the solvent to penetrate into the solid pores, thus enabling oil solubilization. The high temperatures also increase the oil steam pressure making the extraction by the solvent easier. An increase in the pressure makes the solvent penetration into the solid pores easier, providing easier contact among the solvent and the components to be extracted. On the other hand, an increasing temperature at constant pressure leads to a decrease in density.

Table 3
Experimental conditions and extraction yield results for the passion fruit seed oil extraction CO_2 as solvent

Run	Flow rate ($mL\ min^{-1}$)	Pressure (MPa)	Temperature (°C)	Density of CO_2 ($kg\ m^{-3}$)*	Yield (% w/w)
1	1.5	15	40	780.87	2.05
2	1.5	20	40	840.67	3.41
3	1.5	25	40	880.42	6.78
4	1.5	15	50	700.28	1.66
5	1.5	20	50	784.97	4.56
6	1.5	25	50	835.05	7.56
7	3.0	15	40	780.87	2.71
8	3.0	20	40	840.67	11.2
9	3.0	25	40	880.42	18.3
10	3.0	15	50	700.28	2.93
11	3.0	20	50	784.97	17.1
12	3.0	25	50	835.05	18.5

* Angus *et al.* (1976).

Table 4
Global means of the factors studied in the oil extraction from passion fruit seeds with SC-CO_2

Flow rate		Pressure		Temperature	
1.5 $mL\ min^{-1}$	4.3^a	15 MPa	2.34^a	40 °C	8.71^a
3.0 $mL\ min^{-1}$	11.8^b	20 MPa	9.06^b	50 °C	7.40^b
		25 MPa	12.8^c		

Means with equal letters, in the same column, are not significantly different ($p < 5\%$).

4.3. Oil physical-chemical and rheological properties

The seed oil extracted by SC-CO_2 extraction was liquid at room temperature and was golden yellow in color. The specific gravity of the oil was 0.92. The refractive index was 1.4657. The saponification value was 187.9 mg KOH g^{-1}, lower than that obtained by Oliveira *et al.* (2013). The peroxide index value of the oil was 1.25 meq 1000 g^{-1} oil and the value is relatively lower because the oxidization could be avoided during the course of supercritical carbon dioxide extraction. The total acidity, expressed as the acid value, was 2.38 mg KOH g^{-1} oil, lower than tha obtained by Oliveira *et al.* (2013). This value is within the permissible limits for edible oils. The iodine value was 125.8 g I2 100 g^{-1} oil which was lower than that reported by Liu *et al.,* (2009) and Nyanzi *et al.*, (2005).

Regarding the rheological properties, the flow behavior index (n) of passion fruit oil in equation (6) were between 1.204 and 2.065, in the temperature range of 10 to 70 °C, which is considered as a dilatant fluid behavior within the shear rate range from 1.02 to 70 s^{-1}. The dilatant fluid shows an increasing behavior of viscosity with an increase in the shear rate. In a resting situation, the particles are well separated from each other. The increase in the shear rate entails the approximation of the particles, resulting in an increase in the resistance of the moment flow and the viscosity decreased with an increase in temperature. Similar results are reported for corn, canola, sunflower oils (Toro-Vazquez and Infante-Guerrero, 1993), chia seed oil (Ixtaina *et al.,* 2010) and tiger nut oil (Lasekan and Abdulkarim, 2012). The effect of temperature on the viscosity of passion fruit oil was estimated as the Arrhenius-type relationship. The activation energy was 27.7 KJ mol^{-1}.

4.4. Mass transfer behavior

The kinetic model used was successful in describing the mass transfer behavior (Figure 1, Table 5). The analysis showed that extraction occurred at a faster rate. As shown in Table 5, the mass transfer coefficient values increased with temperature at constant pressure and increased with pressure at constant temperature for each flow rate. The same behavior was observed by Özkal *et al.*, (2005) and Adeib *et al.*, (2010). According to Özkal *et al.*, (2005) during the fast extraction, the released oil, on the surface of particles, was

Table 5.
Bayesian estimation of model parameter on mass transfer behavior of passion fruit oil

	Flow rate ($mL\ min^{-1}$)	Pressure (MPa)	Temperature (°C)	k (s^{-1})	Standard deviation
1	1.5	15	40	7.978×10^{-6}	1.994×10^{-7}
2	1.5	20	40	3.542×10^{-6}	1.209×10^{-7}
3	1.5	25	40	1.542×10^{-6}	1.833×10^{-7}
4	1.5	15	50	5.430×10^{-6}	1.092×10^{-7}
5	1.5	20	50	1.527×10^{-5}	1.384×10^{-7}
6	1.5	25	50	2.627×10^{-5}	1.008×10^{-6}
7	3.0	15	40	1.258×10^{-5}	2.892×10^{-7}
8	3.0	20	40	4.357×10^{-5}	3.973×10^{-7}
9	3.0	25	40	8.439×10^{-5}	2.351×10^{-6}
10	3.0	15	50	1.733×10^{-7}	1.746×10^{-9}
11	3.0	20	50	3.949×10^{-5}	6.202×10^{-7}
12	3.0	25	50	8.496×10^{-5}	2.388×10^{-6}

extracted and in this period the transfer resistance was governed by the solubility of oil in solvent, and mass transfer resistance was due to the solvent phase.

5. CONCLUSION

Passion fruit oil was extracted using supercritical CO_2. The effects of temperature, CO_2 flow rate and pressure were investigated. The experimental results have shown that extraction yield increases when temperature, CO_2 flow rate and pressure were increased. The maximum yield was at 3.0 $mL\ min^{-1}$ CO_2 flow rate, 25 MPa and 40 °C. The passion fruit seed oil was rich in unsaturated fatty acids and met the required standards for edible oil. The rheological behavior observed was dilatant fluid and the temperature effect on viscosity was the Arrhenius-type relationship

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